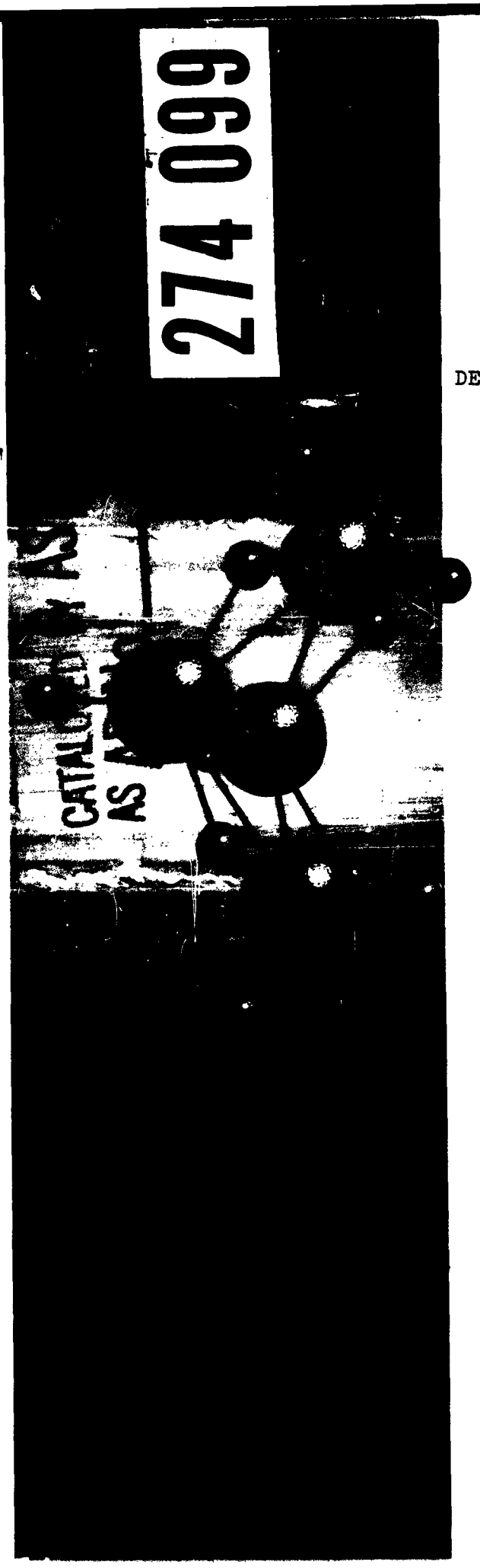

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DETERMINATION AND ANALYSIS OF THE POTENTIALITIES OF THERMAL ENERGY STORAGE MATERIALS

AF 33 (616)-7224

Quarterly Report No. 3

January 1, 1962 - March 31, 1962

GALLERY CHEMICAL COMPANY

DETERMINATION AND ANALYSIS OF THE POTENTIALITIES OF THERMAL ENERGY STORAGE MATERIALS

AF 33(616)-7224

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by

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January 1, 1962 - March 31, 1962

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The work covered by this report was accomplished under Air Force Contract 33(616)7224 but this report is being published and distributed prior to Air Force review. The publication of this report, therefore, does not constitute approval by the Air Force of the findings or conclusions contained herein. It is published for the exchange and stimulation of ideas.

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INTRODUCTION

A program to determine and analyze the potentialities of various thermal energy storage materials is being conducted by Callery Chemical Company. Emphasis at present is on the storage of thermal energy utilizing the latent heat of fusion of the storage materials.

Essentially this program is both a continuation and an expansion of a program started in May 1960 under Contract AF 33(616)7223. Briefly this program consists of:

(1) A continuation of the laboratory experimental program for the measurement of the physical properties of candidate storage compounds or systems at high temperatures;

(2) Design, construction and operation of an experimental unit to study the rate of heat release and movement of the fusion front in compounds or mixtures being considered as heat storage media;

(3) A series of materials of construction--heat storage materials compatibility tests to insure use of the proper container materials for succeeding physical property measurements;

(4) A theoretical analysis to determine the optimum mechanical configuration for the heat storage material with respect to total weight and heat release rate.

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SUMMARY

Compatibility tests to find materials suitable for containing molten energy storage materials have been continued. Fansteel-80, tantalum, molybdenum, graphite and boron nitride have been found to be suitable containment materials for molten lithium metaborate for at least 24 hours in an argon atmosphere. Fansteel-80 has been tested with lithium metaborate for 87.5 hours at 1600°F in an argon atmosphere. No evidence of leakage or contamination of the lithium metaborate was noted. Calcium silicide has been found to be extremely corrosive to all metals including the refractory metals. It has been successfully contained in boron nitride for 36 hours. Ceramics and cermets now appear to be the only potential long duration containment materials for calcium silicide. Compatibility tests with magnesium silicide are inconclusive due to the inability to obtain complete melting of this material.

The heat content measurements on calcium silicide were extended to 1780°F. Attempted measurement of the heat of fusion was not successful due to failure of the platinum sample container. A new attempt to measure heat of fusion will be made using a tantalum capsule since tantalum has shown resistance to attack for short durations.

The thermal conductivity apparatus which failed on the last attempt to measure the thermal conductivity of molten sodium fluoride, has been revised again. A new crucible, fabricated from a new type of graphite has been installed. Tests have shown this new graphite to be compatible with lithium metaborate, which will be the first material used in the revised system.

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The density of molten lithium metaborate has been determined over the temperature range of 1570° to 2040°F. The viscosity of this material has also been determined between 1570° and 1960°F.

Repairs to the high temperature furnace of the heat release measurement apparatus have been completed. Construction of the nickel block calorimeter of the apparatus has been completed and this unit installed. Experimental measurements in this apparatus are expected to start in April.

DISCUSSION OF RESULTS

Materials Compatibility:

The compatibility program is being conducted to find materials suitable for containing molten thermal energy storage materials during experimental measurements. A suitable container material must satisfy the following requirements: (1) no leakage through container walls and (2) no contamination of the storage material due to the container. Energy storage materials included in the program are lithium metaborate, calcium silicide, and magnesium silicide.

Lithium Metaborate

Fansteel 80, tantalum, molybdenum, graphite, and boron nitride are suitable container materials for molten lithium metaborate for at least 24 hours in an argon atmosphere. Haynes metal 25 is not suitable in either air or argon atmosphere.

Fansteel 80 contained molten lithium metaborate at 1600°F for 87.5 hours in argon atmosphere with no evidence of leakage or contamination. As expected, the Fansteel 80 container was completely destroyed by rapid oxidation in air at 1600°F. Tantalum and molybdenum were not attacked by lithium metaborate during a 24 hour test in argon atmosphere. Longer duration tests will be necessary to fully evaluate these materials. A recently received graphite material (impregnated to reduce porosity) for use in the thermal conductivity measurement apparatus was found to be a suitable container material for lithium metaborate for 24 hours in argon. Boron nitride was not attacked by lithium metaborate during 24 hours in argon; however, during 44.5 hours additional exposure in air the capsule walls (15 mil thickness) failed at the liquid interface. The boron nitride is reportedly stable

up to 1800 °F in an oxidizing atmosphere. Further testing in argon is planned. Haynes Metal 25 was severely attacked and leaked badly during 19 hours in air. In argon the Haynes Metal 25 container did not leak in 26 hours; however, the lithium metaborate was contaminated by solution of metallic elements from the container. These tests are summarized in Table 1, and photographs of some of the test containers are shown in Figure I.

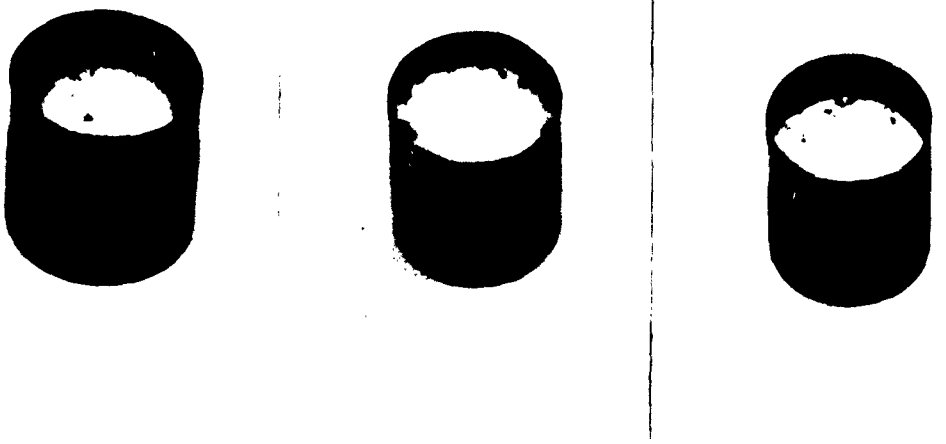
Calcium Silicide

Calcium silicide is extremely corrosive toward metallic construction materials, including the refractory metals. Only tantalum showed any corrosion resistance, and its use is limited to durations of several hours. Calcium silicide has been contained successfully for 36 hours in boron nitride and aluminum oxide. It appears that ceramic and cermet materials are the only potential long duration container materials.

Originally, it was intended to study calcium silicide, CaSi; however, the composition of the commercially available calcium silicide is more nearly $CaSi_2$. Since this commercial grade material has a higher estimated heat of fusion and a lower melting temperature, it appears more attractive as a heat storage material than CaSi.

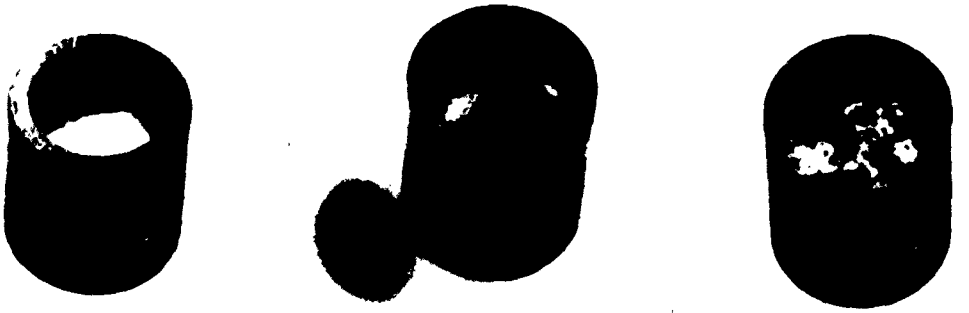
Compatibility tests with $CaSi_2$ are summarized in Table 2; photographs of some of the test containers are shown in Figure II. Nine common construction materials representative of each of the various groups of alloys, were totally destroyed by molten $CaSi_2$. Fansteel 80 and molybdenum were not suitable for even limited service. Tantalum was moderately attacked by molten $CaSi_2$ in 36 hours. The tantalum capsule from Test Number 13 was cleaned of $CaSi_2$ by dilute acid and

FIGURE I



Lithium Metaborate Tests (left to right)

Test Number 19	Fansteel 80	87.5 hours-argon
Test Number 22	Tantalum	24 hours-argon
Test Number 23	Molybdenum	24 hours-argon



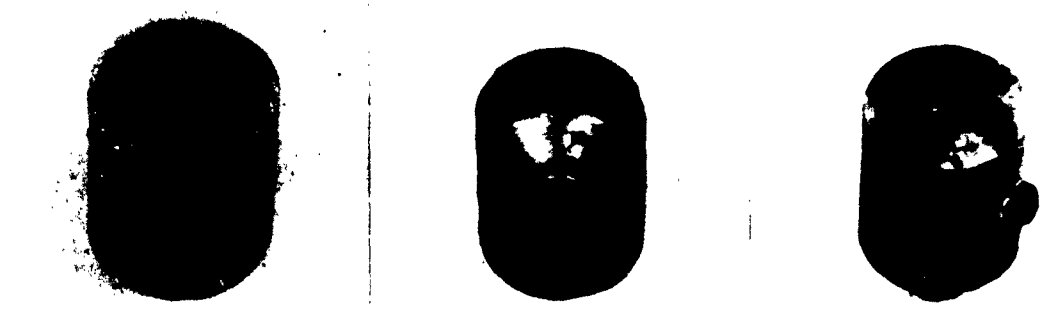
Lithium Metaborate Tests (left and center respectively)

Test Number 24	Graphite	24 hours-argon
Test Number 21	Haynes 25	26 hours-argon

Magnesium Silicide Test (right)

Test Number 3	Inconel 600	32 hours-argon

FIGURE II



Calcium Silicide Tests (left to right)

Test Number 12	Boron Nitride	36 hours-argon
Test Number 13	Tantalum	36 hours-argon
Test Number 14	Molybdenum	36 hours-argon

Calcium Silicide Tests (left to right)

Test Number 8	Inconel 600	16 hours-argon
Test Number 9	Nickel 200	16 hours-argon
Test Number 15	Graphite	38 hours-argon

carefully inspected. It appears that tantalum is suitable for brief measurements such as thermal properties. The impregnated graphite will not be suitable for measuring thermal conductivity of $CaSi_2$ because a chemical reaction occurs forming calcium carbide. Boron nitride was not attacked by $CaSi_2$ in 36 hours; however, it was necessary to use a container with a heavier wall (1/16 inch thick). In an earlier test with a 15 mil wall container cracking of the walls was experienced. It is believed that the cracking was due either to thermal stresses or expansion of the $CaSi_2$ on freezing. Aluminum oxide was also suitable in a 48 hour test.

Although the ceramic type materials are corrosion resistant, they present problems in fabricating containers. Subsequent tests on ceramic and cermet materials will be aimed at finding a material which can be formed into the desired shapes, which can be joined, and which is not brittle. These qualities are requirements for a suitable capsule for heat release rate measurements in the nickel block drop-calorimeter.

Magnesium Silicide

Several tests have been made to determine the compatibility between molten Mg_2Si and container materials. These tests, summarized in Table 3, are somewhat indefinite because of incomplete melting of the Mg_2Si. Although the suppliers claim purities above 98 percent and report a melting point of 2015° F, the material does not melt completely even at 2370° F. The container shown in Figure I is typical of the "lumpy" appearance of the Mg_2Si after a test. X-ray analysis of the magnesium silicide as received shows Mg_2Si, with a major impurity of MgO and a minor impurity of Mg. Elemental analysis showed 77.8 weight percent Mg (theoretical 63.4), 8.5 weight percent Si (theoretical 36.6), and 2.8 weight

percent Fe. The remainder was assumed to be oxygen. In spite of the fact that the bulk of the material does not melt, some corrosion was encountered. Holes developed in the walls of containers made from Inconel 600, Nionel 825, and Nickel 200. It is believed that magnesium is responsible for this effect.

All of the compatibility tests were made using small lumps of Mg_2Si or powder from the lot. One special test was made with a large lump in a porcelain crucible to see if this part of the lot could be melted. This sample also failed to melt at 2190° F. Following this and previous tests, the surface of the container and the top of the furnace tube was covered with a white film. The film is believed to be due to magnesium which has boiled and risen in the furnace tube above the argon atmosphere where it reacted in the oxidizing atmosphere to magnesium oxide.

Special melting point tests using sealed capsules loaded in argon atmosphere also were unsuccessful. The cooling curve was not indicative of a phase change upon cooling from 2370° F.

For the reasons described above, we feel that further work with the commercial grade material would not be worthwhile. Preparations have been reported in the literature by which Mg_2Si is formed by heating stoichiometric amounts of the components in an inert or reducing atmosphere. This preparation technique reportedly gives a powdered product which is more suitable for experimental measurements than the commercially available product which is available only in large lumps. These preparations will be attempted in an effort to obtain a material suitable for property measurements.

Future Plans

1. Compatibility tests for lithium metaborate with tantalum, molybdenum, and boron nitride for 100 hours in argon will be made.
2. Compatibility tests for lithium metaborate with Incoloy 804, Nionel 826, and 446 stainless steel for 24 hours in argon will be made.
3. As a result of the promising resistance of Fansteel 80 to lithium metaborate, long duration tests of 500 to 1000 hours including quantitative evaluation should be obtained.
4. Compatibility tests for calcium silicide ($CaSi_2$) with boron nitride and aluminum oxide for 100 hours in argon will be made.
5. Obtain samples of other ceramic type materials for tests with calcium silicide. Choose those which are least brittle and which are most easily fabricated.
6. Prepare magnesium silicide from the elements.
7. Check the compatibility of sodium fluoride in the impregnated graphite.

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Table 1

LITHIUM METABORATE - MATERIAL COMPATIBILITY TESTS

Test No.	Test Material	Temperature ° F	Atmosphere	Test Duration Hours	Result
19	Fansteel 80	1600	Argon	87.5	No leakage or contamination.
19 a	Fansteel 80	1600	Air	16	Capsule destroyed by atmosphere.
20	Haynes 25	1600	Air	19	Severe attack. Walls badly blistered and split.
21	Haynes 25	1600	Argon	26	Moderate attack. Contents contaminated.
22	Tantalum	1600	Argon	24	No leakage or contamination.
23	Molybdenum	1600	Argon	24	No leakage or contamination.
24	Graphite	1600	Argon	24	No leakage or contamination.
25	Boron Nitride	1600	Argon	24	No apparent attack.
25 a	Boron Nitride	1600	Air	44.5	Attack at interface. Hole in wall.

NOTES:

1. Test 19 a. This test was a continuation of Test 19 using the same container.
2. Test 24. This test container had 0.125 inch wall thickness; all others had 0.015 inch wall thickness.
3. Test 25 a. This test was a continuation of Test 25 using the same container.

Table 2

CALCIUM SILICIDE - MATERIALS COMPATIBILITY TESTS

Test No.	Test Material	Temperature ° F	Atmosphere	Test Duration Hours	Result
1	316 Stn. Stl.	2300	Argon	25	Severe attack. Capsule totally destroyed.
2	Incoloy 800	2300	Argon	27	Severe attack. Capsule totally destroyed.
3	Nionel 825	2300	Nitrogen	28.5	Severe attack. Large pits and holes in wall.
4	Fansteel 80	2300	Nitrogen	27.5	Severe attack. Evidence of chemical reaction.
5	310 Stn. Stl.	2300	Argon	48	Severe attack. Capsule totally destroyed.
6	309 Stn. Stl.	2300	Argon	48	Severe attack. Capsule totally destroyed.
7	Haynes 25	2300	Argon	48	Severe attack. Capsule totally destroyed.
8	Inconel 600	2300	Argon	16	Severe attack. Capsule totally destroyed.
9	Nickel 200	2300	Argon	16	Severe attack. Capsule totally destroyed.
10.	304 Stn. Stl.	2300	Argon	16	Severe attack. Capsule totally destroyed.
11	Boron Nitride	1900	Argon	27	No leakage. Walls cracked.
12	Boron Nitride	1900	Argon	36	No apparent attack.
13	Tantalum	1900	Argon	36	Limited attack.
14	Molybdenum	1900	Argon	36	Severe attack. Holes in walls.
15	Graphite	1900	Argon	38	Chemical reaction to calcium carbide.
16	Fansteel 80	1900	Argon	48	Walls badly attacked.
17	Tantalum	1900	Argon	48	Attack somewhat more severe than in Test 13.
18	Al_2O_3	1900	Argon	48	No apparent attack.

NOTES:

1. Test No. 12, boron nitride container had 0.0625 inch wall thickness.
2. Test No. 15 and Test No.18: graphite and Al_2O_3 containers had 0.125 inch wall thickness.
3. All other containers had 0.015 inch wall thickness.

Table 3

MAGNESIUM SILICIDE - MATERIAL COMPATIBILITY TESTS

Test No.	Test Material	Temperature ° F	Atmosphere	Test Duration Hours	Result
3	Inconel 600	2100	Argon	32	Severe cavitation. Hole in wall.
4	Nionel 825	2100	Argon	32	Severe cavitation. Hole in wall.
5	310 Stn. Stl.	2100	Argon	32	Relatively free of attack.
6	310 Stn. Stl.	2100	Argon	32	Relatively free of attack.
7	Hastelloy X	2100	Argon	32	Relatively free of attack.
8	310 Stn. Stl.	2100	Argon	23	Relatively free of attack.
9	309 Stn. Stl.	2100	Argon	23	Relatively free of attack.
10.	Nickel 200	2100	Argon	23	Severe localized cavitation. Holes in wall.

NOTES:

1. Test 5. This test container contained powdered material from the Var-Lac-Oid lot. All other tests with small lumps from this lot.

2. All containers had 0.015 inch wall thickness.

Calorimetry

The heat content measurements on the sample of calcium silicide were extended to 1780° F. Attempted measurement of the heat of fusion was not successful due to failure of the platinum sample container.

The observed heat content data on the solid calcium silicide are given in Table 4. Occasional repeat measurements were made to determine the effects of prior thermal history. No significant effects were noted, indicating negligible change in the crystal structure of the sample and negligible reaction of the sample with the platinum container.

Attempts to obtain the heat content of the liquid sample and the heat of fusion were stopped by extreme capsule failure. The bottom third of the platinum capsule was almost completely destroyed, apparently by reaction and/or solution in the molten silicide. Exposure of the capsule to the molten sample was estimated from the strip chart record to be of the order of one hour.

Several minor problems arose during the course of the measurements. The nickel alloy wire for supporting the capsule could not be used above about 1650° F and was replaced by B & S No. 28 platinum wire. The pure platinum wire had rather low tensile strength and tended to break occasionally while the sample was equilibrating in the furnace. Platinum-rhodium alloy wire will be tried on future measurements, since the alloys have much greater strength at high temperatures.

Also, at temperatures above 1650° F there was some difficulty with AC pickup in the furnace thermocouple circuit. Positioning the thermocouple so that no contact was made with the furnace tube eliminated this problem.

From separate compatibility tests, tantalum gave good indication of withstanding molten calcium silicide for a sufficient length of time to complete the heat of fusion measurement. A capsule has been fabricated and loaded with a sample in preparation for this measurement.

Table 4

HEAT CONTENT DATA ON CALCIUM SILICIDE

Temperature, °F	$H_t - H_{77}$, BTU/lb
298	41.4
334	48.2
484	76.1
577	94.9
649	108.2
759	128.2
761	125.5
880	149.9
993	169.6
1015	192.4
1103	188.6
1168	221.6
1236	223.6
1319	231.1
1422	249.3
1467	259.9
1666	300.8
1773	328.0

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Physical Properties

Thermal Conductivity

A charge of sodium fluoride was melted in the thermal conductivity furnace and cooled to 900° C (1650° F). During the measurements of the temperature gradients in the sample, the heaters suddenly failed. When the furnace was dismantled, it was found that sodium fluoride had leaked through the graphite crucible walls and corroded the heating ribbon.

A new crucible has been made from another grade of graphite which has been impregnated with a resin, and graphitized to fill the pores. The permeability of the graphite is thus reduced to a very low value, and should eliminate leaking of the molten salt.

In addition, the furnace has been redesigned as follows: The wall thickness of the graphite crucible has been increased to 3/8 inch to reduce the possibility of its leaking. The bottom 8 inches of the crucible rod which acts as a heat sink, has been separated and wound with a separate heating ribbon. Thus this section need not be disturbed if it is necessary to replace the crucible. The crucible section is still heated with two windings for good control of temperature gradients.

Argon gas is now piped in through the bottom of the furnace to provide a better inert blanket around the graphite. The auxiliary heater (formerly the "D.C." heater, but now powered by alternating current) has been redesigned to fill the entire cross section of the crucible cavity except for that space occupied by the thermocouple well. This is to reduce heat loss out the top of the furnace, and to allow more space for insulation between coils of the auxiliary heating ribbon.

An attempt will be made to measure the thermal conductivity of molten lithium metaborate at approximately 1652° F.

Density

The density of molten lithium metaborate has been measured by the method of hydrostatic weighing over the temperature range 1570 to 2040° F. The density values, listed in Table 5, fit the equation

density (grams per milliliter) = $2.251-1.945 \times 10^{-4}$ t (°F)

with a standard deviation of $\pm$ 0.0024.

The density of solid lithium metaborate at room temperature has been measured by displacement of carbon tetrachloride, and a light Kel-F oil. The average value was found to be 2.25 g/ml.

The density data of Shartsis, Cappa, and Spinner [J. Am. Ceram. Soc. 36, 35 - 43 (1953)] for mixtures of lithium oxide and boric oxide can be extrapolated to 50 mole percent lithium oxide. Since this mixture corresponds to the compound lithium metaborate, the density values should be comparable. The extrapolated density at 25° is 2.48 g/ml, and that at 1000° C (1832° F) is 1.85 g/ml, compared to the values 2.25 and 1.895 respectively, found in the present study.

Table 5

DENSITY OF MOLTEN LITHIUM METABORATE

Temperature, degrees Centigrade	Fahrenheit	Density, grams per milliliter
857	1574	1.946
859	1578	1.944
867	1592	1.941
870	1598	1.940
878	1612	1.941
892	1638	1.932
896	1645	1.932
914	1677	1.924
918	1684	1.922
939	1722	1.914
968	1774	1.904
980	1796	1.904
1012	1854	1.896
1016	1861	1.888
1053	1927	1.881
1069	1956	1.868
1070	1958	1.870
1093	1999	1.865
1118	2044	1.850

Viscosity

The viscosity of molten lithium metaborate has been measured over the temperature range 1570 to 1960° F., by the method of counterbalanced sphere. The viscosity values, listed in Table 6, fit the equation

$$\log \text{viscosity (centipoises)} = \frac{9252}{T(^\circ \text{ Rankine})} - 2.260$$

with a standard deviation of less than 10 cp.

A platinum sphere, 1 cm in diameter, was suspended from the left pan of an analytical balance and immersed in the liquid. Upward or downward motion of the ball was produced by changing the weights on the right pan of the balance. The velocity of the ball was measured by timing the motion of the balance pointer across a selected number of scale divisions.

The apparatus was calibrated in the same way using three different liquids as standards. A viscous oil -- Fluorolube T 80, pure glycerine, and a National Bureau of Standards calibrating oil were used at various temperatures to cover a wide range of viscosities. The method is more fully described by Shartsis and Spinner [J. Res. Nat'l. Bur. Standards 46, 176 (1951)].

The rate of motion of the solid platinum ball through the melt was very sensitive to small changes in weights counterbalancing it because of the relatively low viscosity of the melt. For this reason, the experimental errors are large compared with the actual viscosity values. A request has been submitted for quotations on a hollow platinum sphere which would allow more accurate measurements in the low viscosity range.

Table 6

VISCOSITY OF MOLTEN LITHIUM METABORATE

Temperature, degrees Centigrade	Fahrenheit	Viscosity, Centipoises
857	1574	140
867	1592	180
878	1612	165
880	1616	125
892	1638	130
917	1682	100
918	1684	105
939	1722	75
968	1774	105
1016	1861	65
1070	1958	65

Heat Release Rate Measurements:

The objective of this part of the program is to determine and compare heat release rates of candidate thermal energy storage materials as an aid to design optimization of thermal energy storage systems.

The furnace has been rebuilt and tested, and is operating satisfactorily. After a delay of nearly two months from the promised delivery date, the nickel stock for the calorimeter block was received. Machining of the block and installation of the heating element have also been completed, and the apparatus is now being assembled. Following brief tests, experimental measurements will be started during the next month.

Based on the results of the materials compatibility tests with lithium metaborate, only platinum and Fansteel 80 were suitable for the test capsule which contains the storage material. After evaluating quotations received on capsules of Fansteel 80 and platinum the latter was chosen. Since the cost of the Fansteel 80 was near that of platinum (due to the limited forms in which Fansteel 80 is available, a much greater weight is required) and the delivery time for Fansteel 80 was approximately three weeks longer, the platinum capsule was chosen. In addition, the high scrap allowance for platinum and its oxidation resistance at high temperatures are advantages. The capsule has been received and is presently being loaded with lithium metaborate in preparation for experimental measurements.

Since metallurgical advances and increasing demand are expected to make refractory alloys such as Fansteel 80 less expensive and more readily available, this material remains a candidate for larger scale tests and full scale system applications. Therefore, properties of columbium were obtained from the supplier (Fansteel Metallurgical Corporation) and are summarized as follows:

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Columbium has a melting point of 4379° F. Although the oxidation of columbium and its alloys in air at elevated temperatures is a serious disadvantage, there are several properties which make these materials attractive. The density of columbium (8.57 g/cc) is only slightly greater than steel and considerably less than the other refractory metals. Its favorable fabrication characteristics and high temperature mechanical properties also are an advantage. Columbium exhibits excellent room temperature ductility. Columbium increases in tensile strength with cold work such as reduction by rolling, tube drawing, and swaging. Although ductility is reduced in the process, annealing in vacuum above the recrystallization temperature (900-1300° C) restores the maximum ductility. Columbium can be reduced as much as 60 to 80 percent in rolling before annealing is required. To prevent air contamination, the inert gas tungsten arc welding process is used almost exclusively in the fusion welding of columbium. This process provides an inert gas (argon or helium) envelope in the weld area to exclude air from the molten puddle to prevent embrittlement caused by absorption of oxygen and nitrogen. Joints are produced which have the same chemical properties as the parent metal (as opposed to the brazing process). The joint strength equals or exceeds the parent metal strength in most cases, and the joint ductility closely approaches the parent metal ductility.

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